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EFFECT OF ZINC ACETATE CONCENTRATION IN A $\text{Ca}(\text{H}_2\text{PO}_2)_2$ – $\text{Zn}(\text{CH}_3\text{COO})_2$ ELECTROLYTE ON THE STRUCTURE AND PROPERTIES OF PEO COATINGS ON ADDITIVELY MANUFACTURED Ti-6Al-4V ALLOY

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ABSTRACT

This study investigated the effect of the concentration of zinc acetate ($\text{Zn}(\text{CH}_3\text{COO})_2$) in a calcium hypophosphite ($\text{Ca}(\text{H}_2\text{PO}_2)_2$) electrolyte on the formation and properties of coatings produced by plasma electrolytic oxidation on Ti-6Al-4V alloy manufactured by selective laser melting. The plasma electrolytic oxidation process was carried out under constant conditions, while the $\text{Zn}(\text{CH}_3\text{COO})_2$ concentration was varied within the range of 0.1–0.4 mol/L. The coating morphology, elemental composition, phase structure, thickness were studied using light microscopy and scanning electron microscopy with energy dispersive X-ray spectroscopy, X-ray diffraction. The mechanical properties of the coatings were characterized by microhardness and wear resistance tests. Zinc acetate was found to significantly affect the growth and structure of the oxide layers. With increasing $\text{Zn}(\text{CH}_3\text{COO})_2$ concentration, the coating thickness increased from 1.51 ± 0.26 to 5.21 ± 1.27 μm , the zinc content in the surface layer increased, and the morphology changed from an open porous structure to a denser one. According to X-ray diffraction analysis, the titanium substrate made the main contribution to the diffraction patterns, whereas the Ca- and Zn-containing components were in an amorphous, poorly crystalline, or nanodispersed state. The best tribological properties, including the lowest coefficient of friction, were obtained at 0.2 mol/L $\text{Zn}(\text{CH}_3\text{COO})_2$. A further increases in $\text{Zn}(\text{CH}_3\text{COO})_2$ concentration result in greater coating heterogeneity and reduced microhardness of the substrate-coating composite. The results confirm the possibility of controlled modification of plasma electrolytic oxidation coatings for medical applications.

INTRODUCTION

Despite the well-documented limitations of rolling and casting technologies that significantly restrict the design possibilities of Ti-6Al-4V orthopedic implants, this material continues to be widely used and remains a benchmark in trauma and orthopedic surgery due to its favorable combination of high specific strength, corrosion resistance, and biocompatibility, as confirmed by clinical practice (García-Hernández et al., 2025).



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Additive manufacturing technologies, including selective laser melting (SLM), have fundamentally transformed the production of orthopedic and dental implants by largely eliminating the design limitations associated with rolling and casting, enabling the fabrication of geometrically complex, patient-specific structures with controllable porosity that promotes effective bone ingrowth (García-Hernández et al., 2025; Harkin et al., 2018).

The extremely high cooling rates in SLM (10^4 – 10^6 K·s⁻¹) lead to the formation of a metastable acicular α' martensitic structure within columnar prior β grains accompanied by high dislocation density, nanotwins, and V and Al microsegregation, which increases strength but reduces ductility and fracture toughness compared with wrought material, making careful control of the thermal history essential through strategies such as substrate preheating and post-process annealing to decompose martensite and reduce dislocation density (Pedrazzini et al., 2023; Ji et al., 2023).

The use of Ti-6Al-4V implants manufactured by SLM printing in clinical practice immediately after fabrication is associated with several problems related to their surface condition:

1. Increased surface roughness ($R_a = 8$ – $27 \mu\text{m}$) with adhered satellite particles and partially melted powder (Król & Tański, 2016; Foudzi et al., 2024).
2. Lack-of-fusion defects and porosity at the periphery of parts, which act as stress concentrators and initiation sites for fatigue cracks (Gillham et al., 2022).
3. Residual tensile stresses that reduce fatigue life (Vayssette et al., 2020).
4. Biologically inert layers of the native oxide (2–10 nm TiO_2), which do not promote osteoblast adhesion, proliferation, or differentiation (Torres et al., 2018).

These issues do not resolve on their own, making surface post-processing a necessary step if the implant is expected to maintain mechanical reliability, resist corrosion, and support effective biological integration in vivo.

At present, in the Republic of Kazakhstan, electrochemical surface oxidation processes of metals are being actively developed, including anodizing (Serikov et al., 2022; Serikov et al., 2015; Serikov et al., 2016) and plasma electrolytic oxidation, also known as micro-arc oxidation, which has proven to be an effective single-step surface modification method and is currently undergoing active development and application in the Republic of Kazakhstan (Kengesbekov et al., 2026; Sagidugumar et al., 2022; Dogadkin et al., 2025; Sagidugumar et al., 2022).

Plasma electrolytic oxidation (PEO) can be considered a high-voltage evolution of conventional anodizing, in which dielectric breakdown and plasma micro-discharges in aqueous electrolytes generate thick, strongly adherent oxide–ceramic coatings on titanium substrates, transforming the surface from bioinert to bioactive and thereby enhancing the potential for optimized osseointegration of medical implants (Aliofkhazraei et al., 2021).

During PEO in calcium–phosphate electrolytes, high-energy dielectric breakdown and plasma micro-discharges drive the conversion of the titanium surface, resulting in the formation of a heterophase bioactive coating composed of an oxide matrix of anatase and rutile TiO_2 with integrated bioceramic phases such as crystalline hydroxyapatite, tricalcium phosphate, and perovskite-structured calcium titanate (CaTiO_3), whose architecture is governed by the combined action of anodic oxidation of the substrate and plasma-induced processes of deposition, crystallization, and thermal transformation of electrolyte components (Lederer et al., 2019; Yerokhin et al., 2016).

Single-step functionalization of titanium substrates via PEO, incorporating 11 at.% zinc as ZnO , yields highly crystalline multifunctional interfaces that completely inhibit *E. coli* growth, reduce *S. sanguinis* biofilm biomass by over 60%, halve wear-induced mass loss, and accelerate osseointegration through enhanced protein adsorption followed by biomimetic hydroxyapatite formation (Malheiros et al., 2025).

Functionalizing SLM-produced titanium substrates with Zn-containing coatings featuring micro–nano topography yields a clear synergistic effect by enhancing adhesion, proliferation, and osteogenic differentiation of MC3T3-E1 pre-osteoblasts (with increased alkaline phosphatase activity), while simultaneously maintaining antibacterial protection against *Streptococcus sanguinis* and *Fusobacterium nucleatum* through controlled zinc ion release (Wu et al., 2019).

Zn²⁺ ions exhibit broad antibacterial activity by disrupting bacterial membrane integrity, generating reactive oxygen species, interfering with enzyme function, and directly inhibiting biofilm formation, thus acting through multiple parallel mechanisms (Liu et al., 2024).

Zn-modified coatings have a key limitation in that their biological effectiveness is dose-dependent with a narrow therapeutic window, as moderate Zn²⁺ levels activate osteoblasts and promote bone formation whereas excessive concentrations induce cytotoxicity and accelerate bone resorption, making precise control of local concentration and Zn²⁺ release kinetics essential to ensure stable osseointegration (Arcos & Vallet-Regí, 2020).

PEO-based zinc modification of titanium produces a strongly adherent coating with porous micro–nano morphology that enhances wear resistance and corrosion stability (increased impedance modulus and widened capacitive arcs in Nyquist plots) while simultaneously enabling prolonged Zn²⁺ ion release that promotes osteogenic differentiation and mineralization of mesenchymal stem cells, thereby combining mechanical protection and biological activation within a single PEO treatment (Luo et al., 2023).

Calcium hypophosphite (Ca(H₂PO₂)₂)-based systems have been explored for biofunctionalization of β-titanium alloys via suspension particle incorporation and have shown promise, yet the effect of soluble zinc acetate on the structure–property relationships of PEO coatings formed specifically on additively manufactured Ti-6Al-4V substrates remains insufficiently studied, particularly because the substrate is critical and SLM-produced material differs significantly from conventionally processed titanium (Kazek-Kęsik et al., 2015).

The purpose of this study is to determine the effect of the concentration of zinc acetate (Zn(CH₃COO)₂) in a calcium hypophosphite (Ca(H₂PO₂)₂) electrolyte on the formation process, structure, elemental and phase composition, thickness, microhardness, and tribological properties of PEO coatings formed on a Ti-6Al-4V alloy produced by SLM, as well as to determine the optimal electrolyte composition for obtaining functionally promising coatings for biomedical applications.

MATERIALS AND METHODS

The specimens were produced using SLM on an Mlab Cusing R system (Concept Laser, Germany) from Ti-6Al-4V titanium alloy powder. Rematitan powder, which complies with DIN EN ISO 22674 (International Organization for Standardization, 2022), was used as the feedstock. The specimens were discs with a diameter of 10 mm and a thickness of 3 mm. For each processing condition, three specimens were prepared and analyzed.

The SLM process parameters were as follows: laser power – 95 W, scanning speed – 800 mm/s, track spacing – 50-80 μm, and layer thickness – 25 μm. After fabrication, the specimens were heat-treated at 820 °C in a vacuum furnace for 4 h, followed by slow cooling to 250 °C under vacuum. Prior to coating formation, the specimen surfaces were etched in a 50 g/L oxalic acid (H₂C₂O₄) solution in an ultrasonic bath at 90 °C for 1.5 h. After etching, the specimens were rinsed with deionized water and air-dried at room temperature. The Initial series was used as the control and consisted of etched specimens without coating formation. Calcium-phosphate coatings were formed by PEO using a Kikusui PWR2001H programmable DC power supply (Kikusui Electronics Corporation, Japan). The process was carried out under the following conditions: voltage – 200 V, treatment time – 300 s, and current density – 100 mA/cm². For the PEO 2 series, an electrolyte based on calcium hypophosphite Ca(H₂PO₂)₂ was used. For the PEO 3-PEO 5 series, zinc acetate Zn(CH₃COO)₂ was added to the base electrolyte at concentrations of 0.1, 0.2, and 0.4

mol/L, respectively. After PEO treatment, the specimens were rinsed with deionized water and dried at room temperature. Tribological tests were conducted using an Anton Paar TRB3 tribometer (Anton Paar, Austria) in a ball-on-disc configuration under dry friction conditions at room temperature. A 6-mm-diameter steel ball made of 100Cr6 bearing steel was used as the counterbody. The tests were conducted at a normal load of 5 N, a sliding speed of 5 cm/s, and a friction track radius of 2.5 mm. The friction track length was 100 m, and the coefficient of friction was recorded in real time. The microhardness of the coatings was determined by the Vickers method using a DuraScan-20 microhardness tester (EMCO-TEST Prüfmaschinen GmbH, Austria) at a load of 0.245 N and a dwell time of 10 s. Surface morphology and elemental composition were investigated using a JSM-6390LV scanning electron microscope (SEM) (JEOL, Japan) equipped with an INCA Energy Penta FET X3 Energy Dispersive X-ray (EDX) microanalysis system (Oxford Instruments, United Kingdom). The phase composition of the coatings was determined by X-ray diffraction (XRD) using a PANalytical X'Pert PRO diffractometer (PANalytical, Netherlands) with Cu K α radiation ($\lambda = 1.54056 \text{ \AA}$) in the 2θ range from 10° to 90° in θ - 2θ mode with a scanning step of 0.02° . Coating thickness was determined from cross-sectional polished sections using a BX-51 optical microscope with a U-CMAD3 digital camera and a SIAMS 800 image analyzer (Olympus, Japan). The surface was examined in reflected light over the entire specimen area in bright-field mode at a magnification of $\times 1000$; scale bars are provided in all images.

For coating thickness, microhardness, and average coefficient of friction, five measurements were taken on each of three specimens for every condition; therefore, each reported average value was calculated from 15 measurements ($n = 15$). The results are presented as mean $\pm$ standard deviation. Differences among conditions were evaluated for statistical significance using one-way analysis of variance (ANOVA) followed by Tukey's honestly significant difference (HSD) post-hoc test; $p < 0.05$ was considered statistically significant.

RESULTS AND DISCUSSION

Surface morphology and SEM-EDX elemental composition

Figure 1 shows the surface morphology of specimens obtained under various PEO conditions.

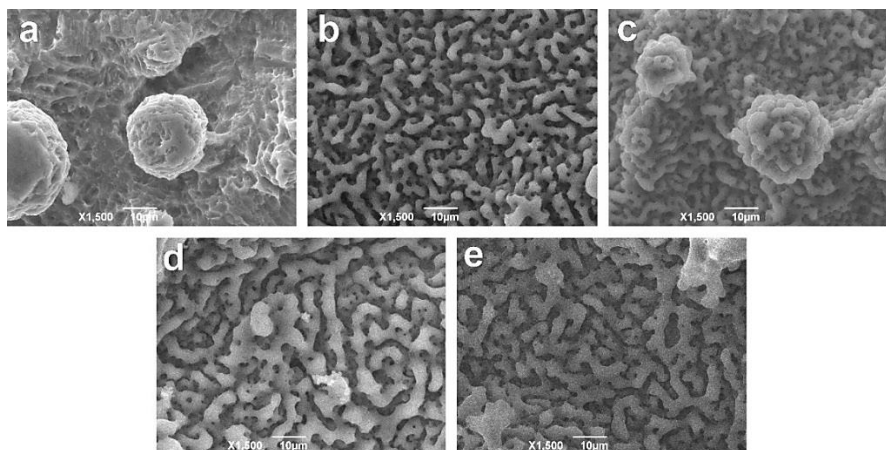


Figure 1. SEM images of the surface of the specimens: (a) 3D-printed initial specimen after etching; (b) 3D-printed specimen treated in 0.5 mol/L calcium hypophosphite electrolyte; (c) 3D-printed specimen treated in 0.5 mol/L calcium hypophosphite electrolyte with 0.1 mol/L zinc acetate; (d) 3D-printed specimen treated in 0.5 mol/L calcium hypophosphite electrolyte with 0.2 mol/L zinc acetate; (e) specimen treated in 0.5 mol/L calcium hypophosphite electrolyte with 0.4 mol/L zinc acetate

Note – compiled by the authors

As shown in Fig. 1a, the surface of the Initial specimens, i.e., the etched specimens without coating, does not show a well-developed porous structure and is characterized by a relatively even relief. Its microtopography appears typical of an additively manufactured material: isolated surface irregularities, traces of partially fused powder particles, and local defects in the form of small depressions and protrusions can be observed. After etching, a substantial fraction of surface contaminants and weakly bonded particles was removed. Still, residual spherical features and traces of the boundaries between melted tracks may remain.

For specimens of the PEO 2 series (Fig. 1b), treated without the addition of zinc acetate, a morphology typical of PEO is formed. The surface consists of a developed porous oxide layer with rounded discharge craters of different sizes and relatively smoothed crater walls. The pores are mostly open and interconnected, forming a branched network of channels. The distribution of craters over the surface is fairly uniform, which may suggest a stable microdischarge regime and relatively homogeneous coating growth. This type of structure has a high specific surface area and can be considered a baseline state for assessing the effect of modifying additives.

For specimens of the PEO 3 series (Fig. 1c), formed with 0.1 mol/L zinc acetate, the coating generally retains the PEO-typical morphology, with a developed porous structure and a system of discharge craters. At the same time, the pores appear to be more evenly distributed over the surface, and their average size tends to decrease. The structure becomes more uniform, which may be related to changes in the microdischarge conditions and to the early stage of zinc incorporation. Local densification and the formation of small agglomerates can be seen in some areas, although the open porous character of the coating is largely preserved.

For specimens of the PEO 4 series (Fig. 1d), formed at 0.2 mol/L zinc acetate, more noticeable morphological changes are observed. The overall open porosity decreases, and the discharge channels become partially blocked. Large discharge craters are still present, but their number is reduced, while the crater walls become thicker and smoother. The structure also becomes more heterogeneous: areas with preserved porosity coexist with regions of a denser oxide layer. This may indicate an intensification of secondary deposition processes, as well as a change in the energy characteristics of microdischarges.

For specimens of the PEO 5 series (Fig. 1e), formed at the highest zinc acetate concentration of 0.4 mol/L, coating densification is most pronounced. The number of open pores and discharge craters decreases markedly, and many of them are either partially or completely sealed. The surface becomes smoother and more compact, with signs of pore filling and the formation of a more continuous oxide layer. Such morphology suggests that deposition and incorporation of zinc-containing products become more pronounced, while the discharge regime is substantially altered. As a result, a denser and less permeable coating structure is formed.

Overall, increasing the concentration of zinc acetate leads to a gradual transition from the classical open porous PEO morphology to a denser and partially sealed oxide layer. This transition is accompanied by a decrease in pore size and pore number, thickening of the crater walls, and, particularly at intermediate concentrations, an increase in structural heterogeneity. The results of the evaluation of the local elemental composition of the coatings using scanning electron microscopy (SEM) with EDX analysis are presented in Figures 2-5 and Tables 1-4. Figures 2-5 show SEM images of the analyzed surface areas and representative EDX spectra, while the quantitative elemental compositions of the selected points are summarized in Tables 1-4. It should be emphasized that EDX provides elemental composition only; phase identification is discussed separately in the XRD section (Fig. 7).

The EDX data for the PEO 2 coating (Table 1) show the presence of O, P, Ca, Ti, Al, and V. The Ca and P signals confirm incorporation of electrolyte-derived calcium-phosphorus components into the oxide coating, whereas Ti, Al, and V are mainly related to the substrate contribution owing to the small coating thickness.

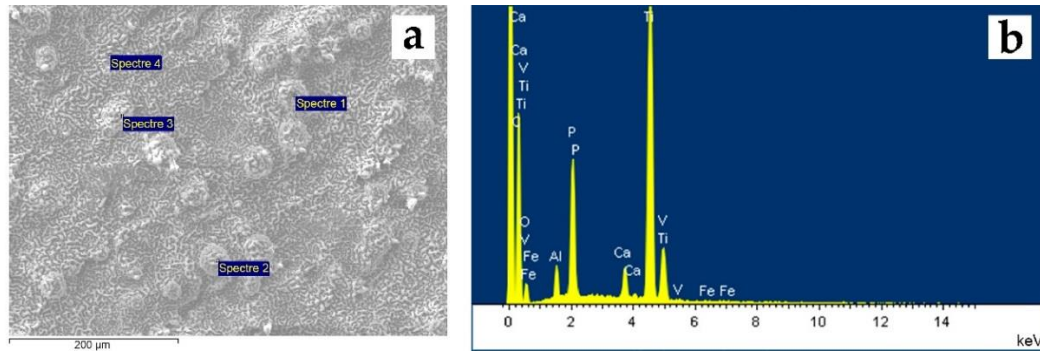


Figure 2. SEM-EDX analysis of the PEO 2 coating formed in a 0.5 mol/L calcium hypophosphite ($\text{Ca}(\text{H}_2\text{PO}_2)_2$) electrolyte: (a) SEM image with EDX analysis points; (b) representative EDX spectrum obtained from the analyzed area, Spectre 1, shown in (a)

Note – compiled by the authors

Table 1. Elemental composition of representative EDX analysis points for the PEO 2 coating shown in Fig. 2a (wt.%)

Spectrum	O	Al	P	Ca	Ti	V	Fe	Total	Ca/P
Spectrum 1	23.36	2.58	12.40	3.42	55.99	2.25	0.00	100	0.27
Spectrum 2	55.54	2.72	6.15	2.43	31.46	1.28	0.41	100	0.39
Spectrum 3	48.95	1.59	15.80	9.71	22.34	1.40	0.21	100	0.61
Spectrum 4	59.09	1.79	10.09	2.66	25.25	1.12	0.00	100	0.26
Average	46.74	2.17	11.11	4.56	33.76	1.51	0.16	100	0.38

Note – compiled by the authors

For the PEO 3 coating formed with 0.1 mol/L $\text{Zn}(\text{CH}_3\text{COO})_2$, the EDX spectrum (Fig. 3b) and Table 2 indicate the initial incorporation of Zn into the coating. The average Zn content is 0.38 wt.%, while Ca and P are also detected, confirming that the coating contains electrolyte-derived components. Because EDX does not identify crystalline phases, the detected Zn should be interpreted as elemental zinc incorporated into the coating rather than as direct evidence of ZnO or other Zn-containing phases.

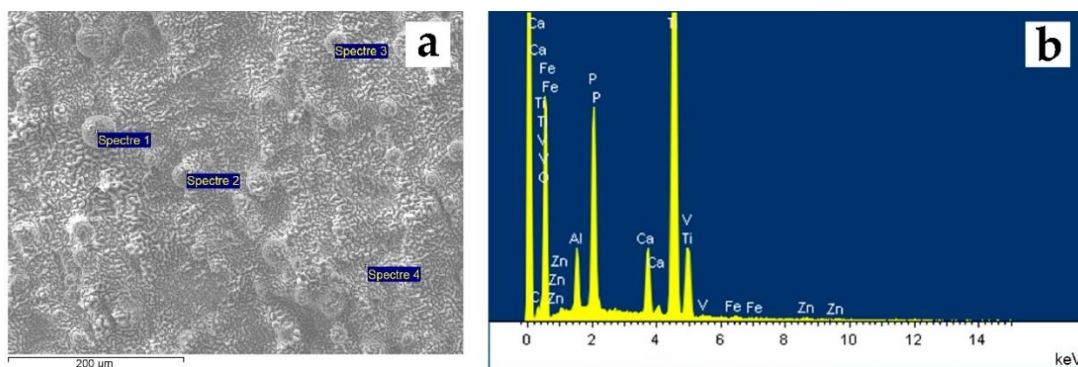


Figure 3. SEM-EDX analysis of the PEO 3 coating formed in a 0.5 mol/L calcium hypophosphite ($\text{Ca}(\text{H}_2\text{PO}_2)_2$) electrolyte with 0.1 mol/L zinc acetate ($\text{Zn}(\text{CH}_3\text{COO})_2$): (a) SEM image with EDX analysis points; (b) representative EDX spectrum obtained from the analyzed area, Spectre 1, shown in (a).

Note – compiled by the authors

The localized presence of Zn in some EDX points suggests non-uniform zinc incorporation at the lowest zinc acetate concentration.

Table 2. Elemental composition of representative EDX analysis points for the PEO 3 coating shown in Fig. 3a (wt.%)

Spectrum	O	Al	P	Ca	Ti	V	Fe	Zn	Total	Ca/P
Spectrum 1	55.33	1.97	6.95	2.91	30.95	1.43	0.00	0.47	100	0.42
Spectrum 2	31.68	2.64	10.86	4.80	47.56	2.46	0.00	0.00	100	0.44
Spectrum 3	45.12	2.28	4.59	2.14	42.04	2.46	0.32	1.05	100	0.47
Spectrum 4	30.01	2.68	12.00	5.46	47.02	2.83	0.00	0.00	100	0.46
Average	40.54	2.39	8.60	3.83	41.49	2.30	0.08	0.38	100	0.45

Note – compiled by the authors

At 0.2 mol/L $\text{Zn}(\text{CH}_3\text{COO})_2$, corresponding to the PEO 4 series, EDX analysis shows a marked increase in Zn and Ca contents (Fig. 4 and Table 3). The average Zn content increases to 2.86 wt.%, and the average Ca/P ratio rises to 0.87. This indicates more intensive incorporation of electrolyte-derived species and a stronger chemical modification of the coating compared with the PEO 2 and PEO 3 coatings.

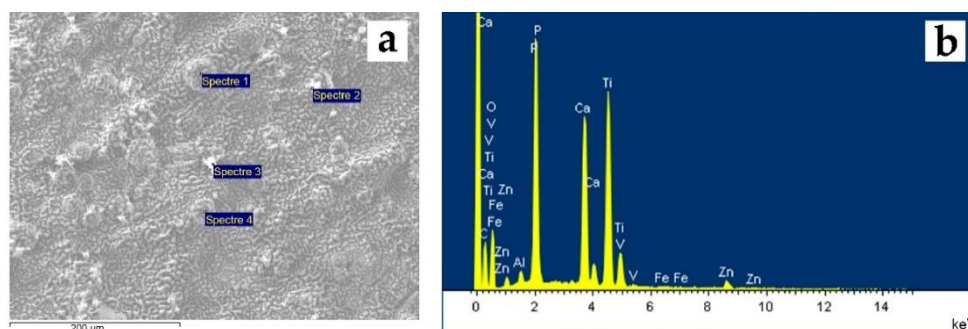


Figure 4. SEM-EDX analysis of the PEO 4 coating formed in a 0.5 mol/L calcium hypophosphite ($\text{Ca}(\text{H}_2\text{PO}_2)_2$) electrolyte with 0.2 mol/L zinc acetate ($\text{Zn}(\text{CH}_3\text{COO})_2$): (a) SEM image with EDX analysis points; (b) representative EDX spectrum obtained from the analyzed area, Spectre 1, shown in (a)

Note – compiled by the authors

The increase in Zn content is accompanied by a lower Ti signal in the analyzed points, which is consistent with a thicker coating and a smaller relative contribution from the metallic substrate.

Table 3. Elemental composition of representative EDX analysis points for the PEO 4 coating shown in Fig. 4a (wt.%)

Spectrum	O	Al	P	Ca	Ti	V	Fe	Zn	Total	Ca/P
Spectrum 1	36.12	0.72	16.44	14.83	26.25	1.50	0.17	3.97	100	0.90
Spectrum 2	49.75	0.78	17.18	17.35	9.79	0.61	0.96	3.58	100	1.01
Spectrum 3	61.24	0.76	15.71	14.55	5.72	0.24	0.17	1.61	100	0.93
Spectrum 4	55.86	1.81	10.82	6.87	20.86	1.20	0.33	2.26	100	0.63
Average	50.74	1.02	15.04	13.40	15.66	0.89	0.41	2.86	100	0.87

Note – compiled by the authors

For the PEO 5 series formed at 0.4 mol/L $\text{Zn}(\text{CH}_3\text{COO})_2$, the EDX spectrum (Fig. 5b) and Table 4 show the highest Zn content among the studied coatings. The average Zn content reaches 8.90 wt.%, although the values vary considerably between points. This confirms intensive but locally heterogeneous incorporation of zinc into the coating.

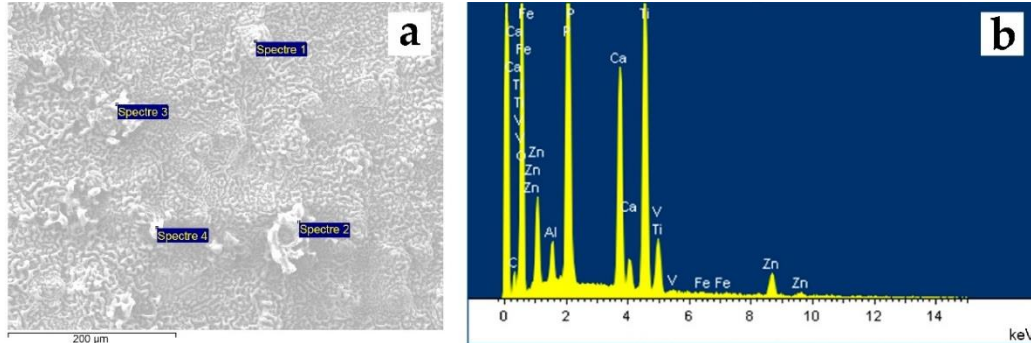


Figure 5. SEM-EDX analysis of the PEO 5 coating formed in a 0.5 mol/L calcium hypophosphite ($\text{Ca}(\text{H}_2\text{PO}_2)_2$) electrolyte with 0.4 mol/L zinc acetate ($\text{Zn}(\text{CH}_3\text{COO})_2$): (a) SEM image with EDX analysis points; (b) representative EDX spectrum obtained from the analyzed area, Spectre 1, shown in (a)

Note – compiled by the authors

The EDX results (Table 4) therefore confirm the formation of a Zn-enriched coating. However, the presence of separate crystalline Zn-containing phases cannot be concluded from EDX data alone and is evaluated in the XRD section.

Table 4. Elemental composition of representative EDX analysis points for the PEO 5 coating shown in Fig. 5a (wt.%)

Spectrum	O	Al	P	Ca	Ti	V	Fe	Zn	Total	Ca/P
Spectrum 1	55.42	0.96	14.05	7.60	15.79	0.85	0.00	4.99	100	0.54
Spectrum 2	53.26	0.79	14.43	13.17	5.18	0.31	0.47	12.39	100	0.91
Spectrum 3	55.89	0.63	16.43	12.47	7.51	0.30	0.00	6.49	100	0.76
Spectrum 4	54.39	0.74	15.20	14.53	2.72	0.00	0.70	11.72	100	0.96
Average	54.74	0.78	15.03	11.94	7.80	0.36	0.29	8.90	100	0.79
<i>Note – compiled by the authors</i>										

With increasing zinc acetate concentration, the degree of Zn incorporation into the coating increases from 0.38 wt.% for PEO 3 to 2.86 wt.% for PEO 4 and 8.90 wt.% for PEO 5. This trend is accompanied by the morphological transition from an open porous structure to a denser and partially sealed coating. The observed changes may be beneficial for corrosion resistance, wear resistance, and biological performance, although these effects should be assessed directly in future studies.

The average EDX-derived Ca/P ratios for the investigated coatings, namely the PEO 2-PEO 5 series, are presented in Tables 1-4 and amount to 0.38, 0.45, 0.87, and 0.79, respectively. In all cases, these values are considerably lower than the stoichiometric ratio characteristic of hydroxyapatite, $\text{Ca/P} \approx 1.67$ (Chocholata et al., 2021). This suggests the formation of calcium-deficient calcium phosphate components rather than stoichiometric hydroxyapatite.

Coating thickness

The microstructure of representative cross-sectional polished sections of the PEO coatings formed on the titanium surface in electrolytes of different compositions is shown in Fig. 6. The coating appears as a thin dark surface layer located between the titanium substrate and the outer region. The coating boundary has a distinctly uneven profile, which is typical of layers formed under PEO conditions. Since the coating thickness varies locally along the surface, five measurements were taken on each of three specimens for every PEO condition ($n = 15$), and the results are reported as mean $\pm$ standard deviation.

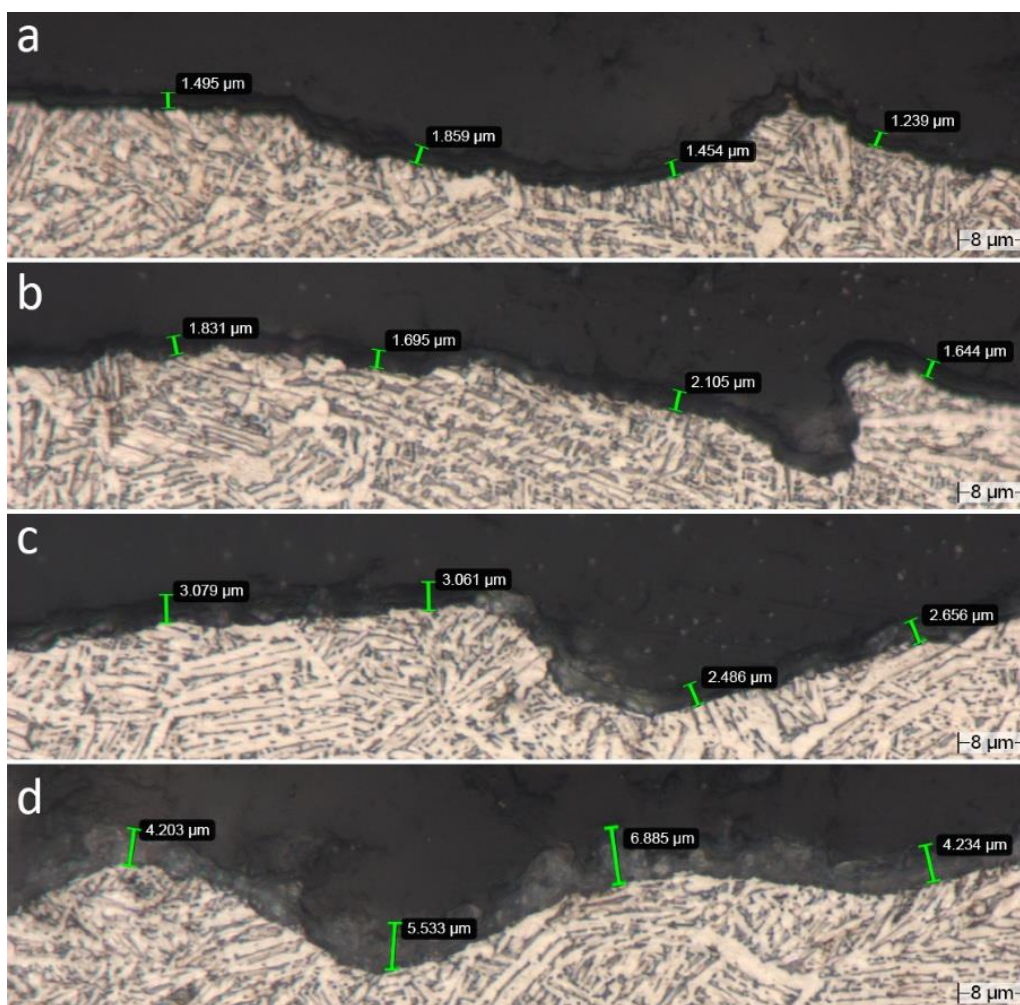


Figure 6. Microstructure of cross-sectional polished sections of PEO coatings on titanium:
(a) PEO 2, electrolyte based on $\text{Ca}(\text{H}_2\text{PO}_2)_2$; (b) PEO 3, $\text{Ca}(\text{H}_2\text{PO}_2)_2 + 0.1 \text{ mol/L Zn}(\text{CH}_3\text{COO})_2$;
(c) PEO 4, $\text{Ca}(\text{H}_2\text{PO}_2)_2 + 0.2 \text{ mol/L Zn}(\text{CH}_3\text{COO})_2$; (d) PEO 5, $\text{Ca}(\text{H}_2\text{PO}_2)_2 + 0.4 \text{ mol/L Zn}(\text{CH}_3\text{COO})_2$.

Note – compiled by the authors

For specimens of the PEO 2 series, obtained in the calcium hypophosphite $\text{Ca}(\text{H}_2\text{PO}_2)_2$ -based electrolyte without the addition of zinc acetate, a thin surface layer is formed. The representative cross-section shows local thickness values ranging from 1.239 to 1.859 μm , while the average coating thickness calculated from all measurements is $1.51 \pm 0.26 \mu\text{m}$. Although the coating is relatively thin, its outer boundary is noticeably uneven, which is likely associated with the specific growth behavior of PEO layers under local micro-arc discharge conditions.

The introduction of zinc acetate, $\text{Zn}(\text{CH}_3\text{COO})_2$, leads to an increase in coating thickness. For specimens of the PEO 3 series, produced with 0.1 mol/L $\text{Zn}(\text{CH}_3\text{COO})_2$, the thickness increases to $1.82 \pm 0.21 \mu\text{m}$, with local values in the representative cross-section ranging from 1.644 to $2.105 \mu\text{m}$. Compared with PEO 2, the average thickness increases by approximately 19%.

When the zinc acetate concentration is raised to 0.2 mol/L, specimens of the PEO 4 series show a more pronounced increase in coating thickness. The measured values in the representative cross-section fall within the range of 2.486–3.079 μm , while the average thickness reaches $2.82 \pm 0.30 \mu\text{m}$. This is about 1.85 times greater than that of the PEO 2 coating.

The maximum thickness is observed for specimens of the PEO 5 series, formed in the electrolyte containing 0.4 mol/L $\text{Zn}(\text{CH}_3\text{COO})_2$. In this case, local thickness values in the representative cross-section range from 4.203 to 6.885 μm , and the average thickness reaches $5.21 \pm 1.27 \mu\text{m}$. At the same time, the scatter of the measurements increases, which may indicate greater thickness heterogeneity and the development of a more pronounced surface relief.

Phase composition (XRD analysis)

The XRD patterns of the initial Ti-6Al-4V specimen and the specimens after PEO are shown in Fig. 7. The Initial specimen is characterized by a set of intense reflections corresponding mainly to the α -Ti phase, along with weaker reflections of β -Ti. According to the XRD data, the α -Ti content is approximately 96.5%, whereas the fraction of β -Ti does not exceed 3.5%. After PEO treatment of specimens of PEO 2–PEO 5 series, the general appearance of the diffraction patterns remains largely unchanged. In all cases, the most pronounced peaks are still associated with the Ti-6Al-4V titanium substrate, which suggests that the contribution from the coating phases is comparatively weak in the recorded diffraction signal.

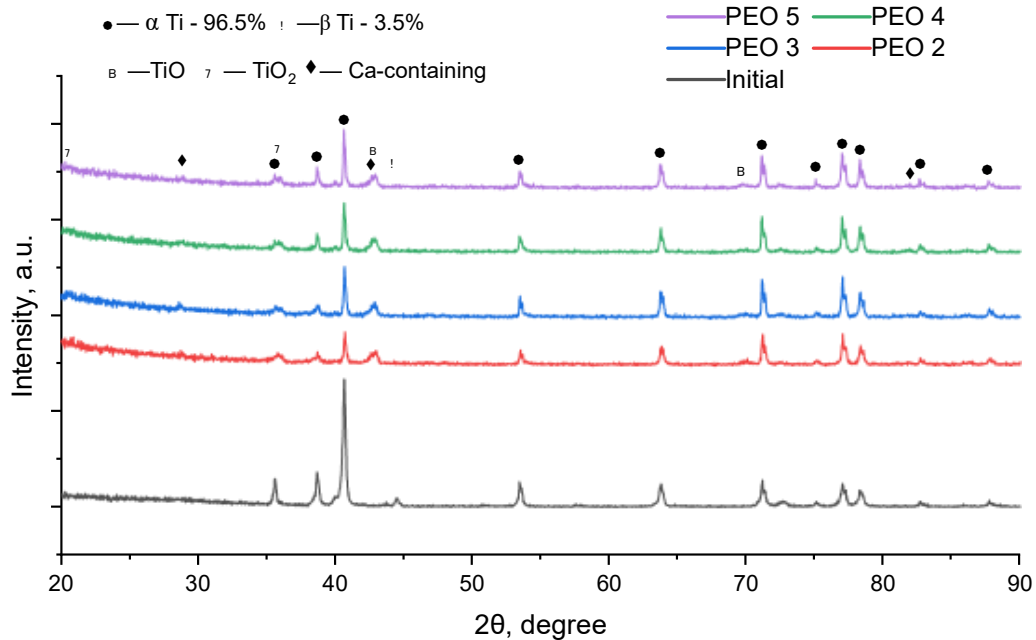


Figure 7. XRD patterns of the initial Ti-6Al-4V specimen and the PEO coatings

Note – compiled by the authors

As can be seen from Fig. 7, the deposition of PEO coatings does not lead to the appearance of intense new diffraction maxima. This suggests that the main contribution to the recorded XRD signal comes from the metallic substrate rather than from the surface oxide layer. This effect is

likely related to the very small thickness of the formed PEO coating: the penetration depth of X-rays exceeds the thickness of the oxide layer, and as a result, the diffraction peaks of α -Ti and β -Ti dominate in all patterns.

Only weak additional reflections are observed in the diffractograms of the coated specimens. These reflections may be associated with the presence of TiO and TiO₂. However, their intensity is very low, and they partly overlap with the strong reflections of the titanium substrate. This may indicate that titanium oxide and titanium dioxide are present in the coating only in small amounts, or that they occur in a poorly crystallized, nanocrystalline, or partly amorphous state.

For specimens of the PEO 2 series, obtained in the Ca(H₂PO₂)₂-based electrolyte, weak indications of calcium-containing compounds may be present. In specimens of the PEO 3, PEO 4, and PEO 5 series, formed in electrolytes containing Zn(CH₃COO)₂ at concentrations of 0.1, 0.2, and 0.4 mol/L, respectively, no clear diffraction maxima that could be unambiguously assigned to separate crystalline zinc-containing phases are detected. It should be noted that the Ca- and Zn-containing components identified by EDX analysis are likely incorporated into the coating in a poorly crystalline, amorphous, or highly dispersed state. For this reason, the available XRD data do not allow one to state with certainty that separate crystalline ZnO phases or complex Ca-Zn-P compounds were formed.

Thus, the XRD analysis indicates that PEO of the Ti-6Al-4V alloy leads to the formation of a thin oxide layer containing small amounts of oxide and Ca/Zn-containing components. However, because of the limited coating thickness, the low content of secondary compounds, and their possible amorphous or nanodispersed state, these phases are barely expressed in the XRD patterns. Accordingly, the diffractograms mainly reflect the phase composition of the Ti-6Al-4V titanium substrate, while the contribution of the PEO coating remains weak.

Tribological properties

Figure 8 shows the tribological test results.

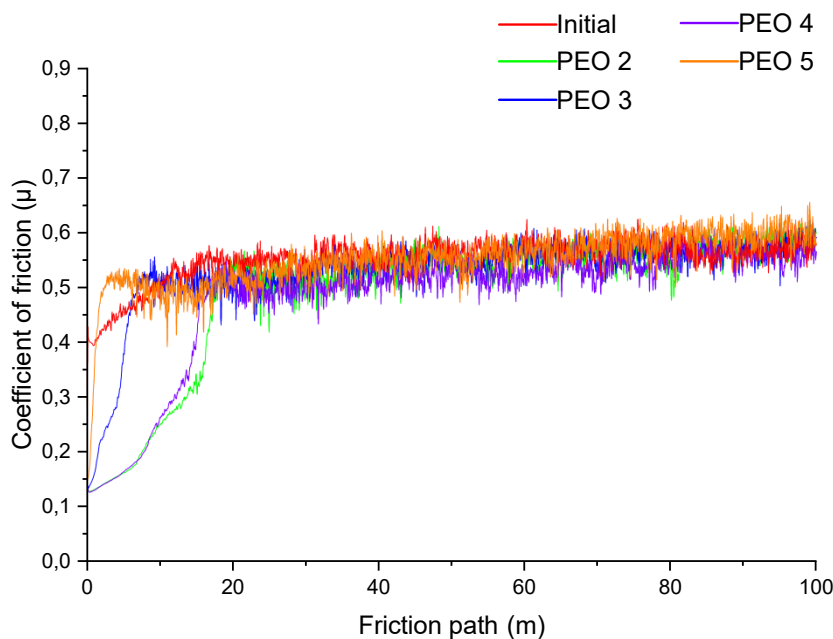


Figure 8. Variation in the coefficient of friction (μ) during dry ball-on-disc sliding as a function of friction path

Note – compiled by the authors

All investigated specimens show typical tribological behavior, with a distinct running-in stage followed by a transition to a quasi-steady-state regime. The duration of the running-in stage depends on the coating composition: for specimens of the PEO 3 and PEO 4 series, it reaches approximately 15-20 m, whereas for the Initial specimens and specimens of the PEO 5 series, stabilization of the friction coefficient is already observed within the first 5-10 m of sliding. The average coefficient of friction, calculated from 15 measurements for each condition, decreases for specimens of the PEO 2 series, reaching $\mu \approx 0.49$, compared with the Initial series, for which $\mu \approx 0.55$. This decrease is likely associated with the formation of a porous oxide layer after PEO treatment. The addition of zinc acetate leads to a non-linear change in tribological behavior. At a concentration of 0.1 mol/L, corresponding to the PEO 3 series, the coefficient of friction increases to approximately $\mu \approx 0.52$, which may be related to structural heterogeneity of the coating. At 0.2 mol/L zinc acetate, corresponding to the PEO 4 series, the lowest friction coefficient is recorded, $\mu \approx 0.48$, along with the smallest amplitude of friction fluctuations. This suggests the formation of the most stable and tribologically favorable coating structure among the studied specimens. With a further increase in zinc acetate concentration to 0.4 mol/L, corresponding to the PEO 5 series, the coefficient of friction rises to about $\mu \approx 0.54$. This effect is probably associated with excessive coating densification, the development of internal stresses, and a reduction in structural stability. For all specimens, a slight increase in the coefficient of friction with increasing sliding distance is observed. This trend may be caused by gradual coating wear and the progressive involvement of the underlying layer in the contact process.

The microhardness values of the coatings obtained by PEO are shown in Fig. 9. For each condition, five Vickers indentations were made on each of three specimens ($n = 15$), and the results are presented as mean $\pm$ standard deviation. For the Initial series, the microhardness was approximately 402 HV. In specimens of the PEO 2 series, the microhardness increased to 420 HV, indicating the formation of a more hardened oxide layer. This increase may be related to the incorporation of calcium- and phosphorus-containing components into the coating, as well as to a higher degree of structural densification.

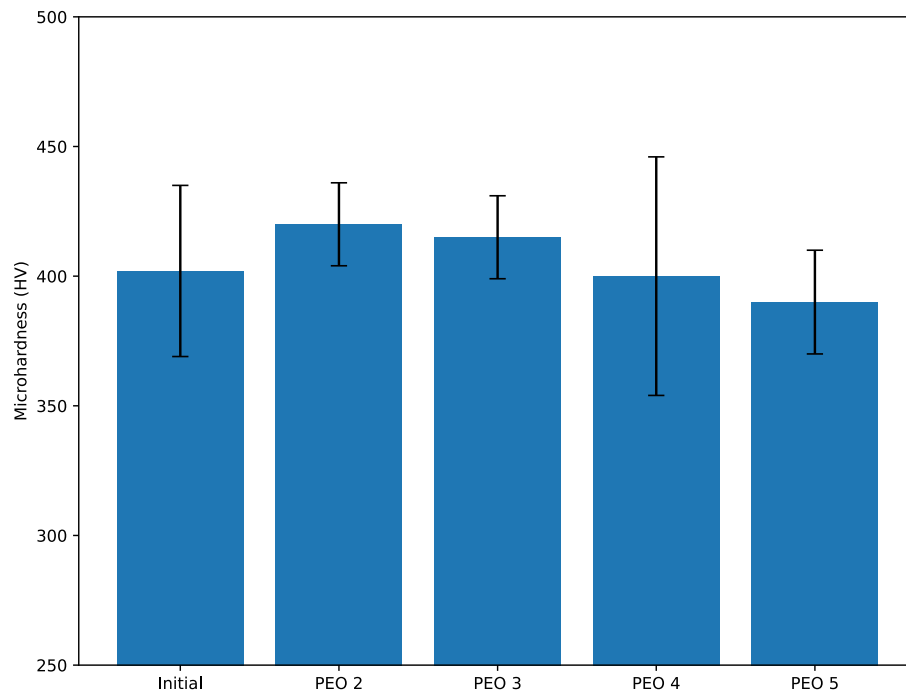


Figure 9. Microhardness of the investigated specimens (mean $\pm$ SD)

The addition of zinc acetate, $\text{Zn}(\text{CH}_3\text{COO})_2$, to the base electrolyte had a somewhat ambiguous effect on the microhardness of the coatings. For specimens of the PEO 3 series, the microhardness was approximately 415 HV, which is close to that of specimens of the PEO 2 series. This suggests that the strengthening effect is largely retained at a low zinc content in the electrolyte.

When the zinc acetate concentration is increased to 0.2 mol/L, corresponding to the PEO 4 series, the average microhardness decreases to approximately 400 HV. This decrease is accompanied by a considerable increase in the scatter of the experimental data. The higher dispersion of the measured values may suggest the formation of a more heterogeneous coating structure, possibly related to increased porosity, local defects, or changes in the phase composition of the oxide layer.

A further increase in the $\text{Zn}(\text{CH}_3\text{COO})_2$ concentration to 0.4 mol/L, corresponding to the PEO 5 series, leads to an additional decrease in microhardness, down to approximately 390 HV. These results suggest that an excessive zinc content in the electrolyte may alter the kinetics of plasma electrolytic processes and promote the formation of a less dense coating with poorer mechanical characteristics.

Thus, the highest microhardness values were obtained for coatings formed in the calcium hypophosphite-based electrolyte without a substantial amount of zinc acetate. The addition of $\text{Zn}(\text{CH}_3\text{COO})_2$ at a low concentration does not appear to have a pronounced negative effect on coating hardness. However, a further increase in the concentration of the zinc-containing additive leads to a gradual reduction in microhardness and to greater structural heterogeneity of the coatings.

It is important to note that no post-test surface characterization (e.g., wear-track profilometry, SEM imaging of the wear scar, or volumetric wear rate determination) was performed in this study. Accordingly, the conclusions about tribological performance are confined to the measured coefficient of friction and cannot be extended to wear resistance or material loss. Post-test characterization is planned for subsequent work and is necessary before any ranking of the coatings by tribological performance can be considered complete.

CONCLUSIONS

The obtained results show that the concentration of zinc acetate in the $\text{Ca}(\text{H}_2\text{PO}_2)_2$ – $\text{Zn}(\text{CH}_3\text{COO})_2$ electrolyte is an effective parameter for controlling the structure and physicochemical–tribological properties of PEO coatings on additively manufactured Ti-6Al-4V alloy. The formation of a Ca-P-Zn-containing oxide layer makes it possible to impart a set of surface characteristics potentially relevant for biomedical implant surfaces, including a developed micromorphology, incorporation of calcium-, phosphorus-, and zinc-containing components, increased coating thickness, and modified tribological behavior. It must be emphasized, however, that no biological characterization (Zn^{2+} ion release kinetics, in vitro cytotoxicity, antibacterial assays) or electrochemical testing (EIS, potentiodynamic polarization) was performed in the present study; accordingly, conclusions regarding biomedical suitability remain inferential and require experimental validation in future work.

The obtained coatings may be regarded as candidate surface layers for further investigation in the context of titanium implants, since they combine the mechanically strong Ti-6Al-4V metallic substrate with a chemically modified surface. The presence of Ca- and P-containing components — albeit in calcium-deficient, amorphous, or nanodispersed form as indicated by the low EDX-derived Ca/P ratios (0.38–0.87) and the absence of distinct Ca-P XRD peaks — may constitute a precursor environment for bioactive behavior, provided that surface crystallization and phase development are promoted in subsequent processing or immersion steps. The incorporation of Zn could further broaden the functional potential of the coating, but

dose-dependent cytotoxicity and ion release kinetics must be characterized before drawing conclusions on biological performance.

From the perspective of biomaterial applications, the PEO 4 series, formed at a $\text{Zn}(\text{CH}_3\text{COO})_2$ concentration of 0.2 mol/L, appears to be the most promising. These specimens are characterized by increased coating thickness, pronounced incorporation of Ca, P, and Zn, and the lowest coefficient of friction among the studied conditions. By contrast, the PEO 5 series, produced at 0.4 mol/L zinc acetate, shows the greatest thickness and the highest Zn content, but this is accompanied by increased structural heterogeneity and reduced microhardness. These features may limit its long-term operational stability.

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STATEMENT ON THE USE OF ARTIFICIAL INTELLIGENCE TECHNOLOGIES:

Artificial intelligence tools, including ChatGPT [OpenAI] and Grammarly AI [Superhuman Platform], were used to assist in rephrasing certain sentences and identifying suitable synonyms to improve clarity and coherence in the scientific discussion. All final content was critically reviewed and edited by the authors to ensure accuracy and integrity.

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